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SPECIFICATION FOR COPPER OXYCHLORIDE, TECHNICAL

(Second Revision)

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SPECIFICATION FOR COPPER OXYCHLORIDE, TECHNICAL

(Second Revision)

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Indian Standard
SPECIFICATION FOR COPPER OXYCHLORIDE, TECHNICAL
(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 31 August 1978, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and Chemical Division Council.

0.2 Copper oxychloride is extensively used for agricultural and horticultural purposes in fungicidal formulations.

0.3 Copper oxychloride is the common name for basic cupric chlorides.

0.4 This standard was first issued in 1959. This was revised in 1969 to incorporate improved requirement for total soluble alkalinity and specifying soluble copper salts in view of the better manufacturing technique becoming available. This revision has been taken up to bring it in alignment with the requirements as stipulated by Central Insecticides Board (CIB).

0.5 In the preparation of this standard due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for copper oxychloride, technical.

2. REQUIREMENTS

2.1 The material shall be in the form of bluish-green coloured homogeneous powder or in such a form that it is possible to easily powder it by crushing under a knife, without a grinding action. It shall be free from foreign matter.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS OF COPPER OXYCHLORIDE, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENTS	METHOD OF TEST, REF TO
(1)	(2)	(3)	(4)
i)	Moisture content, percent by mass, *Max*	1·0	Clause 4.2 of IS : 6940-1973*
ii)	Copper content, percent by mass, *Min*	57·0	Appendix A of IS : 1506-1977†
iii)	Total chlorine content, percent by mass, *Min*	15·5	Appendix A
iv)	Acidity (as H_2SO_4), percent by mass, *Max*	0·05	Clause 11.3.2 of IS : 6940-1973*
	OR		
	Alkalinity (as Na_2CO_3), percent by mass, *Max*	0·5	Appendix B of IS : 1506-1977†
v)	Soluble copper salts, ppm, *Max*	30	Appendix B
vi)	Lead (as Pb), percent by mass, *Max*	0·05	Appendix C
vii)	Arsenic (as As), percent by mass, *Max*	0·05	Appendix D

*Methods of tests for pesticides and their formulations.

†Specification for copper oxychloride, dusting powders (*second revision*).

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to IS : 8190 (Part I)-1976*.

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*Requirements for packing of pesticides: Part I Solid pesticides.

3.2 Marking — The following information shall be marked legibly and indelibly on each container in addition to the information as required under the Insecticides Act and Rules:

a) Name of the material;
b) Copper content, percent, (*m/m*);
c) Name of the manufacturer;
d) Batch number;
e) Net mass of contents;
f) Date of manufacture; and
g) The cautionary notice as worded in Insecticides Act and Rules.

3.2.1 When packed in bags (*see* **3.1**), the pictorial marking for 'USE NO HOOKS. DO NOT PUNCTURE' as prescribed in IS : 1260 (Part II)-1973* shall be stencilled on the bags.

3.2.2 Each container may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian Standard methods for sampling of pesticides and their formulation' (*under preparation*).

NOTE — Till such time the standard under preparation is published, the matter shall be as agreed to between the parties concerned.

5. TESTS

5.1 Tests shall be carried out by appropriate methods as referred to in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of tests.

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*Pictorial markings for handling and labelling of goods: Part II General goods (*first revision*).

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

[*Table* 1, *Item* (iii)]

DETERMINATION OF TOTAL CHLORINE CONTENT

A-1. REAGENTS

A-1.1 Dilute Nitric Acid Solution — 5 percent (*v/v*).

A-1.2 Standard Silver Nitrate Solution — 0·1 N.

A-1.3 Ferric Ammonium Sulphate Solution — 10 percent (*m/v*).

A-1.4 Standard Potassium Thiocyanate Solution — 0·1 N.

A-2. PROCEDURE

A-2.1 Weigh accurately about 0·25 g of the material into a 250-ml Erlenmeyer flask. Add to this about 30 ml of dilute nitric acid solution and shake to dissolve the contents of the flask. Add 20 ml of the standard silver nitrate solution, about 5 ml of ferric ammonium sulphate solution, and titrate the mixture against the standard potassium thiocyanate solution.

A-2.2 Alternatively, direct titration may be carried out potentiometrically.

A-3. CALCULATION

A-3.1 Total chlorine content, percent by mass

$$= \frac{3{\cdot}546 \times (20 - A \times f) \times N}{M}$$

where

A = volume in ml of the standard potassium thiocyanate solution required for the test with the material,

f = correction factor (*see* **A-3.2**),

N = normality of the standard silver nitrate solution, and

M = mass in g of the material taken for the test.

A-3.2 Correction factor (f) is the equivalent value of 1 ml of the 0·1 N potassium thiocyanate solution in terms of 1 ml of the 0·1 N silver nitrate solution:

$$f = \frac{F_1}{F}$$

where

F_1 = normality of the standard potassium thiocyanate solution, and

F = normality of the standard silver nitrate solution.

APPENDIX B

[*Table* 1, *Item* (v)]

DETERMINATION OF SOLUBLE COPPER SALTS

B-1. APPARATUS

B-1.1 Nessler Cylinders — 100 ml capacity.

B-2. REAGENTS

B-2.1 Concentrated Hydrochloric Acid

B-2.2 Concentrated Nitric Acid

B-2.3 Citric Acid

B-2.4 Standard Ammonium Hydroxide Solution — approximately 5 N.

B-2.5 Sodium Diethyldithiocarbamate Solution — Dissolve 1·0 g of sodium diethyldithiocarbamate in 1 000 ml of copper-free water. Filter and keep in an amber bottle and protect from strong light.

B-2.6 Standard Copper Solution — Dissolve 0·392 8 g of copper sulphate pentahydrate in copper-free water and make up the volume to 1 000 ml so that 1 ml of this solution is equivalent to 0·1 mg of copper.

B-3. PROCEDURE

B-3.1 Weigh 25·0 g of sample and extract with cold water and filter into 250-ml volumetric flask and make the volume to 250 ml. The filtrate should be clear. Mix well and take 100 ml out of 250 ml for the estimation of copper.

B-3.2 Add 0·5 ml of concentrated hydrochloric acid and five drops of concentrated nitric acid, boil for about 15 minutes to reduce the volume to about 86 ml and cool. Add 1 g of citric acid and adjust *p*H to 9 by adding standard ammonium hydroxide solution. Transfer the solution to the Nessler cylinder and add 10 ml of sodium diethyldithiocarbamate solution. Compare the yellow colour produced with a control using 3 ml of standard copper solution in place of the material.

B-3.3 The material shall be considered not to have exceeded the prescribed limit if the intensity of the colour produced with the material is not greater than that produced in the control test. Calculate soluble copper salts in terms of parts per million (ppm).

APPENDIX C

[*Table* 1, *Item* (vi)]

DETERMINATION OF LEAD

C-0. GENERAL

C-0.1 Lead is extracted as the dithizonate from a solution of copper oxychloride and estimated photometrically.

C-1. REAGENTS

C-1.1 Dilute Nitric Acid — 50 percent (*v/v*).

C-1.2 Thymolphthalein Indicator — 0·04 percent (*m/v*) in alcohol.

C-1.3 Ammonia-Cyanide Solution — Dissolve 13·3 g of potassium cyanide in 200 ml of water. Add 75 ml of ammonia solution (r.d. 0·9) and make up the volume to 500 ml.

C-1.4 Hydroxylamine Hydrochloride

C-1.5 Tartaric Acid Solution — 50 percent. Dissolve 50 g in warm water and dilute to 100 ml.

C-1.6 Ammonium Hydroxide Solution — r.d. 0·9.

C-1.7 Potassium Hydroxide Solution — 50 percent (*m/v*).

C-1.8 Potassium Cyanide Solution — 13·3 percent (*m/v*).

C-1.9 Dithizone Solution — Dissolve 0·01 g of dithizone in 500 ml of carbon tetrachloride and filter.

C-1.10 Carbon Tetrachloride

C-1.11 Wash Solution — To 25 ml of dilute nitric acid solution, add 2 to 3 drops of thymolphthalein solution and ammonia-cyanide solution till blue in colour. Dissolve 0·1 to 0·2 g hydroxylamine hydrochloride in this solution. (This wash solution should be prepared freshly.)

C-2. PROCEDURE

C-2.1 Weigh accurately 2·5 g of the material and dissolve it in 50 ml of dilute nitric acid. Boil for 2 to 3 minutes and then allow to cool. Transfer the solution to a 250-ml volumetric flask, make up to the mark and mix well.

C-2.2 Pipette out 25 ml of the solution into a beaker, add 5 ml of tartaric acid solution and 4 ml of ammonia solution. (If the solution turns deep blue, decolourize it by adding further tartaric acid.) Cool and add to the decolourized solution potassium hydroxide solution dropwise (2 to 4 ml) until a deep blue colour is obtained. Cool and add potassium cyanide solution (4 to 6 ml) until the solution becomes water-white or light yellow. Add four drops of thymolphthalein indicator solution and adjust the *p*H of the solution as follows:

> If the colour of the solution after adding the indicator is deep blue or dirty green (dependiug on the colour before adding the indicator), add tartaric acid until the colour becomes light blue or light yellow-green. When the solution is faint light blue (light yellow-green), add ammonia solution till a deeper colour is obtained.

NOTE — The colour of the solution depends on the impurities present in the solution.

C-2.3 Add 0·3 to 0·4 g of hydroxylamine hydrochloride to the solution, dissolve it and transfer the solution to a 250-ml separating funnel. Add 25 ml of dithizone solution, shake and allow to settle. Run the lower layer into a second separating funnel. Add 15 ml of dithizone and 10 ml of carbon tetrachloride to the first funnel, shake, allow to settle and run the lower layer into the second funnel. Then add 5 ml of ammonia solution and 10 ml of carbon tetrachloride to the first funnel. Shake as before and examine the lower layer. If colourless or slightly green, run the lower layer into the second funnel. If pink, add 10 ml of dithizone solution, re-shake and run off the lower layer. Wash the aqueous layer with another 10 ml of carbon tetrachloride solution and, if colourless, add it to the previous combined extracts. Discard the aqueous layer in the first funnel.

C-2.4 Add the wash solution (*see* **C-1.11**) to the combined extracts in the separating funnel. Shake and allow to settle. The lower layer should have a distinct pink colour and show no orange tint. Run the lower layer through a dry filter paper into a dry 100-ml volumetric flask. Extract the wash water with 10 ml of carbon tetrachloride and run it into the 100 ml volumetric flask. Make up to the mark with carbon tetrachloride.

C-2.5 Measure the intensity of the pink colour immediately on the spectrophotometer at 520 nm in 1-cm cell using carbon tetrachloride as blank. The colour, finally measured, should be pink, as tendancy to an orange colour indicates the presence of oxidation products. The extinction at 450 nm should not be more than half of that at 520 nm.

C-3. CALCULATION

C-3.1 Calculate the amount of lead present in the solution by plotting the intensity reading on the standard curve previously drawn by using varying concentrations of lead.

APPENDIX D

[*Table* 1, *Item* (vii)]

DETERMINATION OF ARSENIC

D-0. GENERAL

D-0.1 Arsenic is precipitated as the element from a solution of copper oxychloride in hydrochloric acid by sodium hypophosphite. The precipitated arsenic is dissolved in iodine solution and estimated photometrically after reduction of arsene molybdate to ' molybdenum blue '.

D-1. REAGENTS

D-1.1 Dilute Nitric Acid — 50 percent (*v/v*).

D-1.2 Concentrated Sulphuric Acid

D-1.3 Concentrated Hydrochloric Acid

D-1.4 Sodium Hypophosphite

D-1.5 Standard Iodine Solution — 0·02 N.

D-1.6 Sodium Bicarbonate Solution — normal solution.

D-1.7 Sulphuric Molybdate Reagent — Prepared by mixing equal volumes of 13 N sulphuric acid and 6·9 percent (*m/v*) solution of ammonium molybdate.

D-1.8 Sodium Metabisulphite Solution — 5 percent (*m/v*).

D-1.9 Stannous Chloride Solution — 0·2 percent (prepared by diluting concentrated stannous chloride solution with water as required).

D-2. PROCEDURE

D-2.1 Weigh accurately 2·500 0 g of the material and dissolve it in 25 ml of dilute nitric acid. Add 6 to 8 ml of concentrated sulphuric acid when the effervescence, if any, has ceased. Carefully boil the solution until it fumes, to remove organic matter. Cool, add 10 ml of water and refume, taking care to avoid ' spitting '. Cool, and then dissolve the salts by warming with 25 ml of water. Add to the solution 30 ml of concentrated hydrochloric acid and 4 to 6 g of sodium hypophosphite. Warm the solution on a hot-plate until the precipitation is complete and then boil gently for 5 minutes to coagulate the precipitate.

D-2.2 Filter through a No. 4 sintered glass funnel. Wash the beaker twice with 15-ml portions of hot freshly-boiled water, pouring washings through the funnel. Then wash the funnel four times, using 15 ml of hot water each time. Discard the nearly colourless filtrate and thoroughly rinse out the filter flask. Put the filter funnel in position and add 20 ml of iodine solution to the precipitate. Draw the solution very slowly through the filter until all the arsenic is dissolved. (It is easy to determine when the arsenic has been dissolved, since it is always in the form of a dark-brown or black finely-divided powder which shows up well even in traces on the filter.) Again add 20 ml of iodine solution and slowly draw through the filter. Wash the filter with 20 to 30 ml of water until all traces of iodine have been washed out. Transfer this solution to a 100 ml volumetric flask and make up to the mark with distilled water.

D-2.3 Transfer 5 ml of the above solution (or less, arsenic content is high) to a test tube with a mark at 9 ml. Pipette out 1 ml of sodium bicarbonate solution and 2 ml of sulphuric molybdate reagent into the test tube. When the effervescence has ceased, add one to two drops of sodium metabisulphite solution to decolourize the iodine. Make up the volume to the 9 ml mark with water. Mix the solution well and then add 1 ml of stannous chloride solution. Shake the test tube thoroughly.

D-2.4 After 5 minutes, measure the intensity of the blue colour developed on the spectrophotometer, preferably at 840 nm using a 1-cm cell and distilled water as blank. If 840 nm cannot be attained by the spectrophotometer, a lower wavelength of 750 nm may be used.

D-3. CALCULATION

D-3.1 Calculate the amount of arsenic present in the material by plotting the intensity of the colour so obtained on a standard curve previously drawn by using pure arsenic-free copper and arsenic trioxide of analytical reagent grade as standards in concentrations from 0 to 60 µg.

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‡Sales Office in Bangalore is at Unity Building, Narasimharaja Square, Bangalore 560002 22 36 71

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